OGMCOAL - Polymer Testing Information

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Subject: Polymer Testing Information
Attachments: PRESENCE OF POLYMER TEST PROCEEDURE.pdf

Hello Kevin,
Here is some information I received on Polymer Testing from one of the companies we have been in touch with.
Please let me know if you have any questions...
Thanks,

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PRESENCE OF POLYMER TEST PROCEDURE
CRANDALL CANYON MINE
UTAH

For:
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By:

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1. PURPOSE

The purpose of this testing is to provide a means of monitoring the level of residual polymer present within the filtrate released by the geotextile containers.

2. SCOPE OF WORK

This testing procedure applies to all dewatering activity carried out at the Crandall Canyon Mine. Genwal Resources desires a practical and effective method to monitor and test for residual polymer in the filtrate waters prior to returning to the watercourse. An accepted method for determining the presence of polymer is using the flocculation method (a.k.a. China Clay Test). The *"Determination of the Presence of Polymer Using the Flocculation Method"* is attached to this proposed procedure.

It is the intent of the contractor to minimize the use of coagulant and flocculent, and proper management of the polymer feed rates should result in minimal residual product in the filtrate water. However, it is important to monitor the filtrate returning the water to the lagoon to document the potential presence of polymer. A reasonable and effective approach would be to complete the modified qualitative tests frequently on-site and perform the more time consuming and precise quantitative tests on a routine, but less frequent, basis. The qualitative tests procedure would be modified to include visual comparison with control samples prepared with various doses of coagulant and flocculent. The quantitative tests require equipment that is not suitable for a construction site and is best completed in a laboratory setting.

3. MATERIALS, METHODS AND TESTING FREQUENCY

The procedures for completing the testing are attached (Appendix A) and are further documented below:

3.1 Qualitative Tests
Materials and equipment required to perform on-site **qualitative** (modified to also compare with control samples) analysis of released filtrate to be provided by WaterSolve, LLC:

- 250-mL glass jars with lids
- Kaolin Clay Slurry
- Syringes (as appropriate)
- Various control samples containing differing doses of polymers (including no polymer), flocculent and coagulant to perform visual comparisons.

The qualitative tests will be completed at least twice daily (i.e. morning and afternoon) during dredging and dewatering operations.

3.2 Quantitative Tests
On-site materials required for **quantitative** test:

- Sample collection containers will be provided by WaterSolve, LLC for shipment of samples for once weekly quantitative analysis, to be performed at WaterSolve's Laboratory (Grand Rapids, MI). Results will be available within three working days of receipt of the sample.

Quantitative analyses are to be completed once weekly and whenever on-site qualitative tests indicate presence of polymer.

4. REPORTING

Qualitative (daily) testing will be included on the daily log sheets. The notebook with the daily log sheets will be kept on-site at all times and will be available for review upon request.

Quantitative testing will be reported to Dredge America. These results will also be maintained and complied in the job dewatering log on-site.

Appendix A

DETERMINATION OF THE PRESENCE OF POLYMER USING THE FLOCCULATION METHOD

CONTENT

1. PURPOSE

The purpose of this method is to provide a means of qualitatively determining the presence of a flocculent or coagulant within a solution, water sample, etc. Additionally, in cases where the polymer present within a sample is known, this method can be tentatively used as a quantitative measure.

2. SCOPE

This procedure applies to all technical service activity carried out in the Suffolk, Virginia laboratory for the Water Treatments business line.

3. PRINCIPLE

Slurry of kaolin clay is very easily flocculated or coagulated when either a flocculent or a coagulant is present. Therefore, for qualitative purposes, the sample being tested is mixed with kaolin clay slurry, and the effects are visually assessed by comparison to a blank. If polymer is present, significant coagulation or flocculation will be seen from the sample being tested as compared to that seen in the blank.

These principles can also be applied to the quantification of the concentration of polymer present within a sample. However, the exact product present within the sample must be known, and the water used to prepare all solutions must be similar in pH, hardness, etc. to the water present in the sample being tested. Standard solutions containing the known product are prepared at different concentrations. Each solution is then mixed with a kaolin clay slurry, and the settlement time of each is measured. A curve and the equation of the curve are then generated from the obtained results, the settlement time of the unknown sample is measured, and the concentration of the specific product in the unknown sample is calculated by substitution into the obtained equation.

4. PROCEDURE DESCRIPTION

4.1 Hazards and Safety Precautions

The information provided below is not a substitute for the MSDS but is supplementary to it. All users must have read and be familiar with the appropriate manufacturer's MSDS before using the chemicals listed below.

All unknown water samples and polymer solutions should be considered irritants to the skin and eyes.

Contact with calcium chloride powder may cause irritation to the skin, eyes, or respiratory tract.

General laboratory safety procedures should be followed.

4.2 Apparatus and Reagents

Apparatus	Reagents
General Apparatus: 1) 100 mL Glass Mixing Cylinders with Stoppers 2) Syringes (as appropriate) 3) Three Place Top Loading Balance 4) Bottles with Caps (as appropriate) **Additional Apparatus Required for Quantification Procedure:** 1) Laboratory Tumbler 2) Stopwatch 3) Four Place Analytical Balance 4) 110° C Oven 5) 200 mL Volumetric Flasks 6) Graduated Cylinders [500 mL and 1,000 mL]	1) ACS Grade Calcium Chloride 2) Laboratory Grade Kaolin Clay

7) Desiccator 8) Glass Petri Dishes (2) 9) 400 mL Glass Beakers (2)	

4.3 Procedure

Preparation of the Clay Slurry

1. Preparation of a 1% Calcium Chloride Solution

    a. Determine how much 1% calcium chloride will be needed to perform the required testing. Please note that approximately 1.7 mL of a 1% calcium chloride solution is required for each test.
    b. Calculate the required weight of calcium chloride needed to obtain the desired weight of 1% calcium chloride solution using the equation below.

    $$W_1 = \frac{W_2 \ x \ C_2}{C_1}$$

    Where:
    W_1 = Weight of Calcium Chloride Required to Prepare the Solution (g)
    C_1 = Concentration of the Calcium Chloride Being Used (%)
    W_2 = Desired Weight of 1% Calcium Chloride Solution (g)
    C_2 = Concentration of Calcium Chloride Solution Required (1%)

    c. Tare an appropriately sized bottle on a three place top loading balance.
    d. Accurately weigh out the calculated weight of calcium chloride required into the tared bottle. The accuracy of this weight should be ±0.002 g. Add deionized water to the bottle to achieve the desired final solution weight. For example, if 100 g of 1% calcium chloride solution is desired, add 1.000 g of pure calcium chloride to a tared bottle, and add deionized water to achieve a final weight of 100.000 g.

2. Preparation of the Clay Slurry

    a. Determine how much clay slurry will be needed to perform the required testing. Please note that 5 mL of slurry is needed for each test.
    b. Tare an appropriately sized bottle on a three place top loading balance.
    c. Into the tared bottle, weigh out 2 parts laboratory grade Kaolin clay and 1 part 1% calcium chloride solution.
    d. Cap the bottle, and shake vigorously until the contents are homogeneous.

Qualitative Determination of the Presence of Polymer in a Sample

1. Perform a blank as follows. To a 100 mL glass mixing cylinder, add 5 mL of the previously prepared clay slurry and 90 mL of water. Please note that the water used for

this blank should be similar in quality to the water present in the sample which is to be tested with regard to hardness, pH, etc.

2. To a second 100 mL glass mixing cylinder, add 5 mL of the previously prepared clay slurry and 90 mL of the sample being tested.

3. Invert both cylinders three times, and visually assess whether flocculation or coagulation has occurred in the sample being tested by comparing the settlement rate and floc size of the clay in the sample cylinder to the settlement rate and floc size of the clay in the blank sample. Record observations.

Quantitative Determination of the Presence of a Specific Product in a Sample

1. A quantitative determination of the presence of polymer in a sample can only be performed with any accuracy in cases where the exact product present within a sample is known and when the quality of the water being used to prepare all solutions does not vary significantly from the quality of the water sample being tested.

2. Obtain a sample of the product which is known to be present within the sample to be tested.

3. For Dry Products and Solution Products – Prepare a standard stock solution using a sample of the product known to be present and water which is similar in quality to the water present in the sample to be tested.

   a. Calculate the required weight of product needed to obtain 750 mL of a 0.1% solution using the equation below.

   $$W = \frac{C_1 \, x \, V}{C_2}$$

   Where:
   W = Weight of Product Required (g)
   C_1 = Desired Solution Concentration (0.1%)
   V = Desired Amount of Solution (750 mL)
   C_2 = Approximate Solids Content of Product

   b. Using a three place top loading balance, tare a 1,000 mL plastic bottle.
   c. Into a tared plastic bottle, weigh out the calculated weight of product required to two decimal places.
   d. Using a 1,000 mL graduated cylinder, measure out 750 mL of make-up water, and add the water to the bottle.
   e. Cap the bottle immediately, and shake the bottle and its contents vigorously to disperse the polymer.

f. If the product is a flocculant, place the prepared solution on a laboratory tumbler for 2 hours. If the product is a coagulant, place the prepared solution on a laboratory tumbler for 10 minutes.
g. Obtain two glass Petri dishes, and check to make certain that the dishes are clean. Mark the dishes as required.
h. Place the dishes in a 110° C oven for ten minutes.
i. Transfer the dishes directly from the oven into a desiccator, and allow the dishes to cool completely.
j. Using a four place analytical balance, weigh one of the Petri dishes to four decimal places, and record the weight in mg as weight D.
k. Using a 60 mL syringe, measure out 60 mL of stock solution, and place this solution into the Petri dish. Make sure the measured volume is free of air bubbles.
l. Repeat Steps j and k for the remaining dish.
m. Place both dishes in a 110° C oven overnight.
n. Transfer the dishes directly from the oven into a desiccator, and allow the dishes to cool completely.
o. Weigh each dish to four decimal places, and record each weight in mg as weight S.
p. Calculate the total solids content of each using the recorded weights and the equation below.

$$\text{Total Solids Content (mg/L)} = \frac{S - D}{0.06}$$

Where:
S = Weight of Dish + Sample in mg
D = Weight of Dish in mg

q. Average the results of the two tests.
r. Obtain two 400 mL glass beakers, and check to make certain that the beakers are clean. Mark the beakers as required.
s. Place the beakers in a 110° C oven for ten minutes.
t. Transfer the beakers directly from the oven into a desiccator, and allow the beakers to cool completely.
u. Using a four place analytical balance, weigh one of the beakers to four decimal places, and record the weight in mg as weight D.
v. Using a 500 mL graduated cylinder, measure out 500 mL of make-up water, and place this water into the beaker.
w. Repeat Steps u and v for the remaining beaker.
x. Place both beakers in a 110° C oven overnight.
y. Transfer the beakers directly from the oven into a desiccator, and allow the beakers to cool completely.
z. Weigh each beaker to four decimal places, and record each weight in mg as weight S.
aa. Calculate the solids content of each water sample using the recorded weights and the equation below.

Solids Content of Water (mg/L) = $\frac{S-D}{0.50}$

Where:
S = Weight of Beaker + Sample in mg
D = Weight of Beaker in mg

bb. Average the results of the two tests.
cc. Calculate the actual concentration of the prepared stock solution using the results from Steps q and bb and the equation below.

Actual Solution Concentration (mg/L) = P – W

Where:
P = Average Solids Content, in mg/L, of the stock solution
W = Average Solids Content, in mg/L, of the make-up water

4. For Liquid Dispersion or Emulsion Grade Products – Prepare a standard stock solution using a sample of the product known to be present and water which is similar in quality to the water present in the sample to be tested.

   a. Perform a % solids determination as outlined in Tech-SOP 1369.
   b. Calculate the required weight of product needed to obtain 750 mL of a 0.1% solution using the equation below and the results obtained from Step a.

$$W = \frac{C_1 \, x \, V}{C_2}$$

Where:
W = Weight of Product Required (g)
C_1 = Desired Solution Concentration (0.1000%)
V = Desired Volume of Solution (750 mL)
C_2 = Solids Content of the Product (obtained from % solids determination)

   c. Using a 1,000 mL graduated cylinder, measure out the quantity of make-up water necessary to make a total solution volume of 750 mL, and add the water to a 1,000 mL plastic bottle.
   d. Using a four place analytical balance, tare an appropriately sized syringe.
   e. Into the tared syringe, weight out to two decimal places the required amount of product. Record to four decimal places the exact weight of product in the syringe.
   f. Inject the contents of the syringe into the bottle containing the make-up water. Cap the bottle immediately, and shake the bottle and its contents vigorously to disperse the polymer.
   g. Place the prepared solution on a laboratory tumbler for 2 hours.

h. Calculate the exact concentration of the prepared stock solution using the recorded weight and the equation below.

$$C_1 = \frac{W \ x \ C_2}{V}$$

Where:
W = Weight of Product (g)
C_1 = Solution Concentration
V = Volume of Solution Prepared (750 mL)
C_2 = Solids Content of the Product (obtained from % solids determination)

5. Prepare a series of standard solutions from the stock solution.

a. Using a 20 mL syringe, measure out 20 mL of the stock solution, and place this stock solution into a 200 mL volumetric flask.
b. To the volumetric flask, add approximately 100 mL of make-up water, and swirl to mix. Once sufficiently mixed, add make-up water until the 200 mL mark is reached. Stopper the flask, and invert the flask several times to mix the solution. This solution is one tenth as concentrated as the stock solution.
c. Using a 20 mL syringe, measure out 20 mL of the solution prepared in Step b, and place this solution into a 200 mL volumetric flask.
d. To the volumetric flask, add approximately 100 mL of make-up water, and swirl to mix. Once sufficiently mixed, add make-up water until the 200 mL mark is reached. Stopper the flask, and invert the flask several times to mix the solution. This solution is one tenth as concentrated as the solution prepared in Step b.
e. Using a 20 mL syringe, measure out 20 mL of the solution prepared in Step d, and place this solution into a 200 mL volumetric flask.
f. To this volumetric flask, add approximately 100 mL of make-up water, and swirl to mix. Once sufficiently mixed, add make-up water until the 200 mL mark is reached. Stopper the flask, and invert the flask several times to mix the solution. This solution is one tenth as concentrated as the solution prepared in Step d.
g. Using a 20 mL syringe, measure out 20 mL of the solution prepared in Step f, and place this solution into a 200 mL volumetric flask.
h. To the volumetric flask, add approximately 100 mL of make-up water, and swirl to mix. Once sufficiently mixed, add make-up water until the 200 mL mark is reached. Stopper the flask, and invert the flask several times to mix the solution. This solution is one tenth as concentrated as the solution prepared in Step f.
i. The solutions prepared to this point should consist of the following nominal concentrations: 1,000 mg/L, 100 mg/L, 1 mg/L, and 0.1 mg/L. The exact concentrations are dependent on the exact concentration of the stock solution as determined in either Step 3-cc or Step 4-h.

6. Measure the settlement time of each solution.

a. To a 100 mL glass mixing cylinder fitted with scale tape, add 5 mL of the previously prepared clay slurry and 90 mL of the 10 mg/L standard.
b. Invert the cylinder three times, and using a stopwatch, measure the time taken for the mudline formed by the flocculated clay to travel from the 3 cm mark on the cylinder to the 7 cm mark on the cylinder. The stopwatch is to be started when the mudline reaches 3 cm and stopped when the mudline reaches 7 cm. This constitutes the settlement time, in seconds, given by the 10 mg/L standard.
c. Repeat Steps a and b for the nominal 1 mg/L standard, for the nominal 0.1 mg/L standard, and for the water sample being tested.

7. Determine the quantity of the specific product in the sample being tested.

a. Using the obtained settlement times for the three standards, plot a graph of settlement time vs. concentration.
b. Fit this data with the best fit curve, and obtain the equation of the generated curve from the software.
c. Substitute the obtained settlement time from the unknown sample for Y in the obtained equation. Solve this equation for X. The resulting value obtained for X is the concentration of the specific product, in mg/L, within the sample.

5. ABBREVIATIONS

MS	Management System
PO	Procedure Owner
MSDS	Material Safety Data Sheet
WP	Name and abbreviation of the Segment Water and Paper Treatment

6. REFERENCES, DOCUMENTS AND RECORDS

References or Related Documents

Document	Document Description
TS&D/Flocculants/General/1436	SOP from former Quality Management System

List of changes

Rev.	Date	Cause of change	compiled by
00	12/11/03	Issue of first edition of MS documents for Segment WP	Stan Barto

7. ATTACHMENTS

Not Applicable

8. DISTRIBUTION

Standard operating procedures for the WT technical service department in Suffolk are available on the company network under the following path:

Siteshare/Sales Dept/Tech Service/Admin and General/Methods